ASSESSMENT OF SPONTANEOUS COMBUSTION OF SOME INDIAN COALS USING DIFFERENTIAL THERMAL ANALYSIS

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Assessment of Spontaneous Combustion of Some Indian Coals Using Differential Thermal Analysis

Dissertation submitted in partial fulfilment

of the requirements for the degree of

Bachelor of Technology

in

Mining Engineering

by

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based on research carried out

under the supervision of

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May, 2016

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Supervisors' Certificate

This is to certify that the work presented in the dissertation entitled "ASSESSMENT OF SPONTANEOUS COMBUSTION OF SOME INDIAN COALS USING DIFFERENTIAL THERMAL ANALYSIS" submitted by *Dibyanshu Shekhar Bhoi,* Roll Number 112MN0427, is a record of original research carried out by him under our supervision and guidance in partial fulfilment of the requirements of the degree of *Bachelor of Technology* in *Mining Engineering*. Neither this dissertation nor any part of it has been submitted earlier for any degree or diploma to any institute or university in India or abroad.

Prof. D.S.Nimaje

Assistant Professor

Declaration of Originality

I, *Dibyanshu Shekhar Bhoi*, Roll Number *112MN0427* hereby declare that this dissertation entitled *"ASSESSMENT OF SPONTANEOUS COMBUSTION OF SOME INDIAN COAL USING DIFFERENTIAL THERMAL ANALYSIS"* presents my original work carried out as an undergraduate student of NIT Rourkela and, to the best of my knowledge, contains no material previously published or written by another person, nor any material presented by me for the award of any degree or diploma of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the dissertation. Works of other authors cited in this dissertation have been duly acknowledged under the sections "Reference". I have also submitted my original research records to the scrutiny committee for evaluation of my dissertation.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

May 16, 2016
NIT Rourkela

Dibyanshu Shekhar Bhoi

Acknowledgment

I wish to express my significant appreciation and obligation to Prof. D. S. Nimaje, Department of Mining Engineering, National Institute of Technology Rourkela for presenting the topic and for his rousing direction, helpful feedback and profitable proposal all through the venture work.

I am also grateful to Prof. M.K. Mishra, Head of the department for helping me in completion of my project in all respects. I am also thankful to Mr. A.K. Mohanto, Mr. D. Amat and other staffs of Department of Mining Engineering for their assistance and help in carrying out different experiments in the laboratories.

I would also like to express my thankfulness to the authorities of MCL and CCL for their assistance in collection of coal samples.

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Abstract

Self-oxidation of coal is one of the major causes for disastrous accidents in coal mines in leading coal producing countries like China, India and Australia. Hence, study of spontaneous combustion of coal is of utmost importance as it not only causes loss of life and property but also a loss of non-renewable power source, i.e., coal. In case of self-combustion of coal, it is very difficult to control the fire in the mine itself. It is important to have knowledge about the susceptibility of coal to spontaneous heating to know the degree of proneness and take steps to prevent any mishap or loss of life, coal reserve, raising concerns about economic aspects of mining etc.

This B.Tech dissertation deals with assessment of susceptibility of some Indian coals to spontaneous combustion using curves obtained from differential thermal analysis of 20 coal samples collected from different coal mines of MCL and CCL, both opencast and underground.

The determination of intrinsic properties as well as susceptibility of the samples was done using following experimental techniques:

- Proximate analysis
- Differential thermal analysis.

It was found that only the Transition temperature determined from the DTA curve is not sufficient in determination of proneness of coal to spontaneous combustion but slopes of the Stage IIA, stage IIB and overall slope at Stage II and further studies based on the proximate analysis of coal and area under the DTG curve obtained from differential thermal analysis (DTA-TG).

Keywords: *Self oxidation; spontaneous heating; degree of proneness; differential thermal analysis; transition temperature.*

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Chapter 1

INTRODUCTION

1.1. Background

About 28.9% of world's energy consumption comes from coal and 41.3% of electricity generated in world making coal as the ultimate source of energy. In India it accounts for 70% of primary commercial energy supply making it the dominant energy source of the country. Spontaneous combustion is the major cause of mine fires in Indian coalfields, despite various preventive measures. The cause of spontaneous combustion is widespread and takes various studies and theories to zero in on the exact cause, but over the years many examinations have been done to find out the proneness of coal to auto oxidation that leads to spontaneous combustion. Knowing the proneness of coal of the one can take preventive measures to avoid spontaneous combustion and protect live and property, safeguard environment and economic aspects of mining [1].

A number of methods have been adopted over the years to identify the proneness of coal to spontaneous combustion. The experiments and studies done to find out the susceptibility of coal to spontaneous combustion are Wet oxidation potential, Crossing point temperature, Differential thermal analysis, Olpinski index, Flammability temperature etc. The proneness of self-heating also accounts for the incubation period of the coal. Incubation period is the time interval between exposure of coal to open air and catching fire by itself. Thus the studies help in finding out the appropriate incubation period for coal of a mine thus helping the planning engineers to plan out the advancement of a face well in advance without any problem or take proper precautionary measures beforehand. The experimental methods used in the project are proximate analysis and Differential thermal analysis [1].

1.2. General

Coal is an important resource for a country as it is not only used as a power source but also for many industrial purposes like refining metals. India is the world's third largest coal consumer behind China and the United States; and the share of coal in India's electricity mix has been rising. In 2015 India's coal consumption was estimated at 790 million tonnes (or 516 million tonnes of coal equivalent), around 10 per cent less than the United States (IEA 2014f).

Thermal coal accounts for around 85 percent, or 665 million tonnes, of India's coal consumption. Metallurgical coal (80 million tonnes) and lignite (45 million tonnes) make up the balance. In the NPS, India's coal consumption is projected to more than double, increasing at an average annual rate of 2.8 per cent, to 1170Mt or 765 Mtce. India is projected to overtake the United States as the world's second largest coal consumer before 2020, and remain the second largest consumer after China for the remainder of the projection period [2].

The power sector accounts for more than 70 percent of India's coal use and supported a five-fold increase in coal use in electricity generation over the past few decades. As such, the power sector is clearly central to the coal outlook in India. India's steel production has increased by around 25 per cent over the past five years to around 83 million tonnes in 2014. The cement industry, the second largest globally after China, is also a major coal user, accounting for around 5 per cent of total coal use. Other industrial sectors, including brick manufacture, consume small quantities of coal. . The top coal producing states are Orissa, Jharkhand, Chhattisgarh, West Bengal, Bihar, Andhra Pradesh and Madhya Pradesh [2].

Fires in coal mines are a major problem nowadays in the mining industry all over the world. Fire in mines can be divided into two types near surface fires and deep fires. In near surface fires, fire reaches to the surface and consumes the oxygen required from the atmosphere and in deep fires the oxygen is obtained from ventilating air in the mines. Self-oxidation takes place because the permeability of coal allows the air to oxidise coal few centimetres inside coal but does not have enough air to remove the heat created which accumulates to increase the temperature and thus causes fire. An incident of spontaneous fires also takes place in opencast colliery mainly near the stock pile area [3].

Spontaneous heating of coal occurs auto oxidation (increase in temperature due to exothermic internal reactions), followed by thermal runway (auto oxidation which rapidly accelerates to high temperatures) and finally, ignition. When coal is exposed to air, some of its exposed parts absorb free oxygen at a faster rate than others and oxidation results with the evolution of several gases such as CO, CO_2, water vapour and others. This takes place at ambient temperatures which leads to open fires [4].

Mine fires not only poses a problem because of fire but also as consequences of fires like evolution of toxic gases, roof fall, damage to support system, haulage systems and some others. These can be prevented by proper investigation of coal seam at regular intervals and precautionary measures should be taken in advance. The first fire was reported in Raniganj

Coalfields in 1865. Many fires have been reported from Jharia and Raniganj coalfields having superior non-coking coal. Many studies were carried out to understand the mechanism of spontaneous heating of coal to adopt some preventive measures against these fires and to run the process of production of coal without any interruption and to save life, property and environment. A reasonable experimentation of the region is required so that the mine planning committee can plan out a mine beforehand keeping in mind the incubation period of coal of nearby mines. Many methods have been used by researchers all over India to predict the proneness of coal to spontaneous combustion like, Wet oxidation potential difference method, Crossing point temperature method, Differential Scanning Calorimetry Technique, Critical Air Blast and Differential thermal analysis [4].

1.3. Objective of the project

The objective of the project is to correlate the results of proximate analysis of coal and differential thermal analysis of coal to predict the susceptibility of coal to spontaneous combustion.

1.4. Methodology

To achieve the above said objective, the following methodology was adopted.

1.4.1. Literature review

Gathering information of all the past works done by scientists and researchers in the aforementioned field.

1.4.2. Sample collection and preparation

Collection of samples from different mines coving few coalfields of India and preparing the samples as per the Indian standards.

1.4.3. Experimentation

The following experiments were carried out on collected coal samples to achieve the above-said objective:

(i) Proximate analysis of coal

(ii) Differential thermal analysis

1.4.4. Analysis

Regression analysis was carried out between proximate analysis and DTA of coal samples collected.

Chapter 2

LITERATURE REVIEW

2.1. Coal mine fires

Mine fires mainly take place in coal mines but fires due to spontaneous combustion also takes place in metal mines for example pyrite mines. The main causes of mine fires are spontaneous combustion, blasting in mines, electrical failure and explosion of gases, spontaneous combustion being the major cause. The process of self-combustion of coal in coming with contact to atmospheric oxygen without any involvement of actual fire is termed as spontaneous combustion [5].

There are two types of cause of fire in mines namely, Endogenous and Exogenous fires.

2.1.1. Endogenous fires [5]

1. Pyrite fires: Like coal, pyrite also under favourable conditions react with atmospheric air to liberating heat whose accumulation over time leads to auto oxidation and thus fires. Tendency of pyrite to self-oxidise is not like that of coal but increase with increase in carbonaceous matter present. There are occurrences when pyrites with 5 - 6 % C and 10 – 12 % S have caused fire due to spontaneous combustion.
2. Bacterial origin: Decaying timber also leads to accumulation of heat with may lead to spontaneous combustion over time.

2.1.2. Exogenous fires [5]

One of the main reasons of exogenous mine fires is electricity. It may initiate from short circuit of electric wires used, heat generated out of machineries, flames from ignition, candles or explosion while blasting and ignition of inflammable gases and materials like timber, oil or wastes, at the time of processing of sulphide ores or fires from surface.

2.2. MECHANISM OF SPONTANOEUS HEATING

The self-oxidation of coal takes place through a series of complex physio-chemical processes which consists of events like absorption of atmospherical oxygen, formation of coal-oxygen complexes and decomposition leading to evolution of heat. Due to the presence of a vast variety of constituents in coal, it makes the oxidation itself is a very complex process with overlapping and intersecting chemical processes making it difficult to separate out the exact cause. The rate of oxidation at ambient environment provides an idea about the proneness of coal to auto oxidation. Due to presence of several constituents in coal a very long stable chemical oxidation takes place. The noticeable compositional, elemental and structural variations reveal that the reaction of oxygen with solid coal is a time dependent dynamic method.

The response environment is heterogeneous inherent in light of the fact that two mass stages, solid and gas are available and extraneous in light of the fact that different auxiliary changes brought by the response, influence the general coal reactivity. Because of the porosity of coal it retains the fluids or gasses or the arrangement of gas/fluid, which is known as sorption. At the point when collection limits at first glance, freeing warmth and rate of entrance of air is irrelevant then it is considered as adsorption yet in the event that uniform infiltration in the majority of the strong happens, then it is called absorption. The retentions is dependably an endothermic marvel and begins from the surface of the strong and devours warmth of the strong for entrance. The vitality at first glance is constantly low. The technique where physical powers like between sub-atomic fascination are appropriate, are known as physical adsorption or Van der Wall's adsorption, however when agent drive needs to break the synthetic way of the compound is called chemisorption or actuated adsorption. This physical adsorption is overwhelming at low temp though substance adsorption is low at low temp while rate of response increments with expansion in temperature. Coal gets warmed up on engrossing oxygen, whose decay marvel can be communicated in the accompanying way. Oxidation is moderate beneath 50°C and quickens above 50°C, however above 80°C, a time of relentless state is kept up, most likely because of the expulsion of dampness of coal. The expulsion of oxides of carbon happens from 120°C. The collaboration of oxygen with coal quickens quickly up to 180°C and warm disintegration begins between 180°C to 220°C. Self-managed procedure of burning starts in the middle of 220°C to 275°C with extremely fast ascent of temperature until the ignition point is accomplished [6].

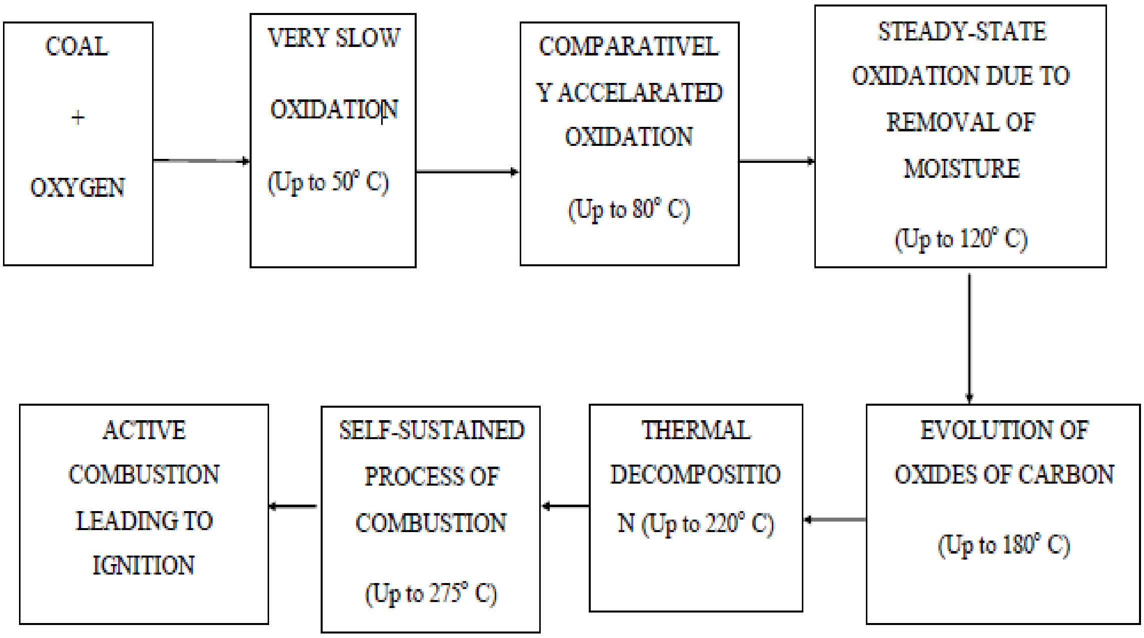

Figure 2.1: Sequence of stages in spontaneous combustion of coal [6].

2.3. FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

There are various reasons for spontaneous combustion and each and every reason plays an important role in the process of auto-oxidation to some extent. The reason for such a widespread condition is because of the presence of a number of constituents in the coal itself. The major reasons enumerated by various researchers are as followed.

Table 2.1 Factors affecting spontaneous combustion of coal [6]

Extrinsic Factors	Intrinsic factors
Bacteria	Chemical constituents
Barometric pressure	Mineral matter
Coal seam and surrounding strata	Moisture
Method of working	Particle size and surface area
Oxygen concentration	Petrographic composition
Roadways	Porosity
Temperature	Pyrites
Timbering	Rank and petrographic constituents
Ventilation system and air flow rate	Volatile matter

2.4. THEORIES OF SPONTANEOUS COMBUSTION OF COAL

Some of the theories laid down by researchers for spontaneous combustion of coal are:

2.4.1 Pyrite theory

Pyrite present in coal as impurity acts as one of the major causes for auto oxidation as when pyrite, present in considerable amount reacts with atmospheric oxygen give out heat and by products of volume more than the reactants hence increasing the porous nature of coal and increasing the heat produced. The reaction involved in the process is given by [6]:

$$2Fe\ S_2 + 70_2 + 16H_2O \rightarrow 2\ H_2SO_4 + 2\ Fe\ S0_4.7H_2O + 316\ kcal$$

2.4.2. Bacterial Theory

The involvement of bacterial processes in spontaneous combustion of coal is not studied to great extend but still it cannot be ruled out. Fires in haystacks and grains stored over time is mainly due to the by-products of bacterial decomposition. However, there is no vital proof to authenticate or cast out this theory [6].

2.4.3. Humidity theory

The amount of heat freed via auto oxidation of coal is substantially less than the measure of heat required for expelling water from the coal. In the event that the evaporation can be impelled toward the beginning of heating, then the temperature of warming would lessen. When it is reviewed that water is an oxidation result of low temperature oxidation of coal, the above framework well clarifies other conceivable wellsprings of CO and CO2 in low temperature response amongst coal and oxygen [7].

Mukherjee and Lahiri (1957) proposed the following mechanism of the reaction between water and coal at 100°C. (Brackets indicate chemi-sorption):

$$H_2O \rightarrow (H)\ (OH) \rightarrow (H_2)\text{-}(0) \rightarrow (H_2) + 0_2$$

$$C + 0 \rightarrow (CO) \rightarrow CO$$

$$(CO) + (0) \rightarrow (CO_2) \rightarrow CO_2$$

2.5. LITERATURE REVIEW

National status:

Panigrahi and Sahu (2004) collected coal samples from different coal seams in various mines for the assessment of spontaneous heating susceptibility by experimental techniques and found that coal seams having transition temperature in the range of 122°C to 140°C are highly susceptible to spontaneous combustion, coal samples with transition temperature in the range of 140°C to 170°C are moderately susceptible and poorly susceptible afterwards [7].

Mohalik et al. (2009) presented the review of application of three thermal techniques viz; differential thermal analysis (DTA), thermogravimetry (TG) and differential scanning calorimetry (DSC); for studying the susceptibility of coal to spontaneous heating and fire. It also critically analyses the experimental standards adopted by different researchers, while applying these techniques in studying thermal behaviour of coal samples. The paper also presents the future direction of research in this subject area [8].

Saikia et al. (2009) coal samples were collected from Ledo and Tikak collieries of Makum coalfield Assam and coal samples were characterized by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) techniques. On heat treatment in air atmosphere up to 800 °C, 20–27% weight loss occurs due to removal of various volatile materials. DTA results indicate the chemical reactivity of the coal sample initially at 80–110 °C due to loss of water, and two other major reactions at around 420 and 530 °C due to primary and secondary volatization [9].

Nimaje et al. (2010) of all the test methods created thermal studies play a critical and predominant part in surveying the auto oxidation of coal. They made an outline of thermal studies completed by various analysts over the world for determination of auto oxidation of coal and uncovered that part of accentuation on test methods is important for developing proper procedures and viable arrangements ahead of time to avoid event and spread of flame [10].

Pattanaik et al. (2011) collected coal samples from different coal seams of Chirimiri coalfields and experimented upon proximate analysis and differential thermal analysis and results were recorded. The coals of higher ranks were less prone to spontaneous combustion whereas coals of lower ranks were more prone to spontaneous combustion [11].

International status:

Ottaway (1982), Cumming & McLaughlin (1982), Rosenvold et al. (1982), and Elder (1983) Coals tested were from the United Kingdom and the United States. Reasonably good agreement was reported with the standard proximate analysis determinations of all the coals. However, a closer examination of the data from these studies reveals the agreement is not within precision limits set by the standards.

Gouws et al. (1988) gave three characteristics on a differential thermal analysis thermogram (i.e., the crossing-point temperature, stage II exothermicity gradient and the transition temperature to high-level exothermicity) are generally believed to be indicative of the self-heating propensity of coal. A new index was developed and applied to 58 coals, enabling known dangerous and safe coals to be identified [12].

Clemens et al. (1990) used DTA at varying temperatures to analyze the reactions between dried low-rank coals and oxygen that provided an immediate and sharp exothermic response. The exotherm increased with increasing temperature and was mostly found in highly susceptible coals to spontaneous combustion. A second exotherm was seen below 120 °C temperature after 15-20 min [13].

Jose, et al. (1996) used differential thermal analysis (DTA) as a method to study the self-heating behaviour of fresh and oxidized coals. Oxidation was performed in air at 200°C for periods of up to 72 h. As the rank of the coal increases, both the self-heating and the end of combustion temperatures also increase. The total heat loss (area under the DTA curve) increases with the rank of the coal. An increase in the self-heating temperature, a decrease in the temperature of the end of combustion and a decrease in total heat flow were observed as a consequence of coal oxidation. A relationship between the total heat loss and the calorific value as determined using the ASTM standard method is pointed out [14].

Chapter 3

EXPERIMENTAL INVESTIGATION

3.1. EXPERIMENTAL TECHNIQUES

The following experiments were carried out to find out the proneness of coal to spontaneous combustion.

1. Proximate Analysis
2. Differential Thermal Analysis

3.2. SAMPLE COLLECTION AND PREPARATION

3.2.1. Sampling

Sampling is the process of collection of a small portion of a whole for experimental purposes so that the small portion represents the properties of the whole substance [15].

Various types of sampling are:

1. Chip sampling
2. Channel sampling
3. Drill-hole sampling
4. Bulk sampling

For the fulfilment of the experiment, the commonly adopted channel sampling method was carried out.

Channel sampling:

In underground mines, the exposed faces of an ore body are sampled from wall to wall at intervals along the face. This is performed by channel sampling. The face to be sampled should be free from dust and altered ore. Loose materials can be removed by brushing or by washing with pressurised water. In case of irregularities in face, it should be smoothened to reasonable extend. By using a hammer and a pointed moil, a groove of about 30 cm wide and 10 cm deep at right angles to the hanging wall and foot wall is cut. The chips are collected in a plastic sheet section-by-section and then put into sample bags and duly labelled [16].

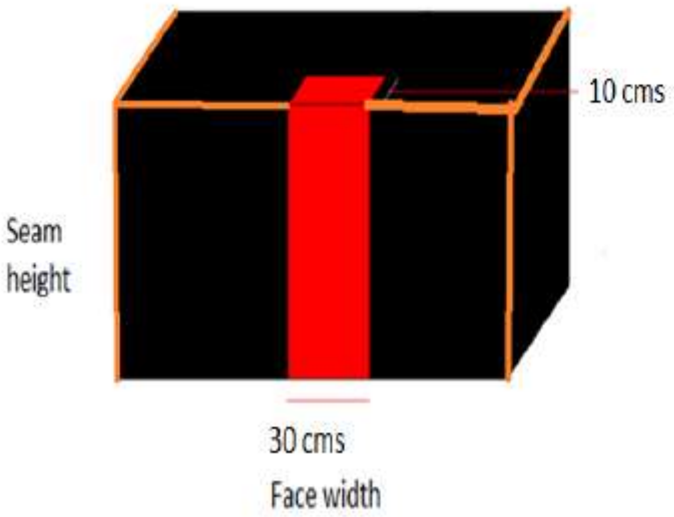

Figure 3.1. Channel sampling [17]

3.2.2. Sample preparation

The samples obtained from the mines by channel sampling are crushed in the laboratory as per the experimental requirements. The crushed samples are then sieved to required sizes and stored in air tight zip packets. The packets are stored in air tight containers for further use in experimentation. Samples were prepared according to IS 436 Part 1, Section 1-1964 and IS 436 Part II-1965 [17].

3.3. EXPERIMENTATION

3.3.1. Proximate analysis

Proximate analysis of coal sample is done to find out the percentage of distribution constituents like (i) moisture, (ii) volatile matter, (iii) ash and (iv) fixed carbon content. The method determined by IS (Indian standard) 1350 (Part-I) – 1984 was followed for determining the proximate analysis of the sample [18].

(i) Determination of moisture content:

Coal is always associated with moisture in one or the other way. It may be external moisture whose main cause is the way coal is handled while mining or while transporting, or it may be intrinsic which is bonded with the chemical structure of coal. External moisture may be removed by air drying but intrinsic moisture can only be removed by heating the coal up to around 100°C.

Procedure:

1. About 1g of finely powdered -212 micron air-dried coal sample is weighed in a silica crucible and then placed in an electric hot air oven and is maintained at 110°C
2. The sample is heated in furnace for 90mins and taken out and places in desiccator for 15 mins.
3. The sample is then weighed and percentage of moisture is found out.
4. The moisture is calculated as per the following

$$\text{Moisture \%} = \frac{Y-Z}{Y-X} * 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots [3.1]$$

Where,

X - Weight of empty crucible, in grams (g)

Y - Weight of crucible + coal sample before heating, in grams (g)

Z - Weight of crucible + coal sample after heating, in grams (g)

(ii) Determination of volatile matter:

Coal consists of various volatile matters like hydrogen, carbon monoxide, methane and other hydrocarbons, ammonia, some organic sulphur and some incombustible gases, such as carbon dioxide and water vapour which plays a major role in spontaneous combustion of coal.

Procedure:

For determining the volatile matter a special volatile matter silica crucible (38 mm height, 25 mm external diameter and 22 mm internal diameter) was used.

1. An empty silica crucible was heated to about 800°C for an hour.
2. It was weighed after that.
3. After taking approximately 1 g of coal sample it was heated to 925°C.
4. After heating it exactly for 7 minutes, the crucible was removed, cooled in open air and then in a desiccator and weighed again.

$$\text{Volatile matter \%} = \frac{Y-Z}{Y-X} * 100 - M\% \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots [3.2]$$

Where

X = weight of empty crucible, in grams (g)

Y = weight of crucible + coal sample before heating, in grams (g)

Z = weight of crucible + coal sample after heating, in grams (g)

M%= Moisture percentage

(iii) Determination of ash:

Ash is the residue substance left after heating a coal sample. It is not present in coal but results after heating of the mineral matter present in the coal. Thus mineral matter and ash are two different things.

Procedure:

1. Weight of the empty crucible is taken.
2. Approximately 1g of coal sample is weighed in the crucible and put in a furnace at 450°C for 30 minutes and then the temperature of the furnace is raised to 850°C for 60 minutes.
3. The crucible is then taken out and placed in a desiccator and weighed

$$\text{Ash \%} = \frac{Z-X}{Y-X} * 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots [3.3]$$

Where,

X= weight of empty crucible (g)

Y= weight of coal sample + crucible before heating (g)

Z= weight of coal sample + crucible after heating (g)

(iv) Determination of fixed carbon content:

There is no separate experimental procedure for determination of fixed carbon content. It is determined by subtracting the moisture, ash and volatile matter percentage from cent percent.

$$\text{Fixed Carbon \%} = 100 - \text{M\%} - \text{A\%} - \text{VM\%} \ldots\ldots\ldots\ldots\ldots\ldots [3.4]$$

3.3.2. Method for determination of susceptibility of coal to spontaneous combustion:

Differential thermal analysis

Differential thermal analysis (DTA) is carried out to represent the physical property of substance under experimentation as a function of temperature. In DTA sample of the substance and an inert substance are heated simultaneously in identical conditions and the change in temperature between the two is graphed with respect to temperature or time. The components of a DTA apparatus are sample and reference holder, furnace, temperature controller and output monitor. The temperature is increase by the temperature controller at a constant rate and the temperature difference of inert reference and sample with time is plotted.

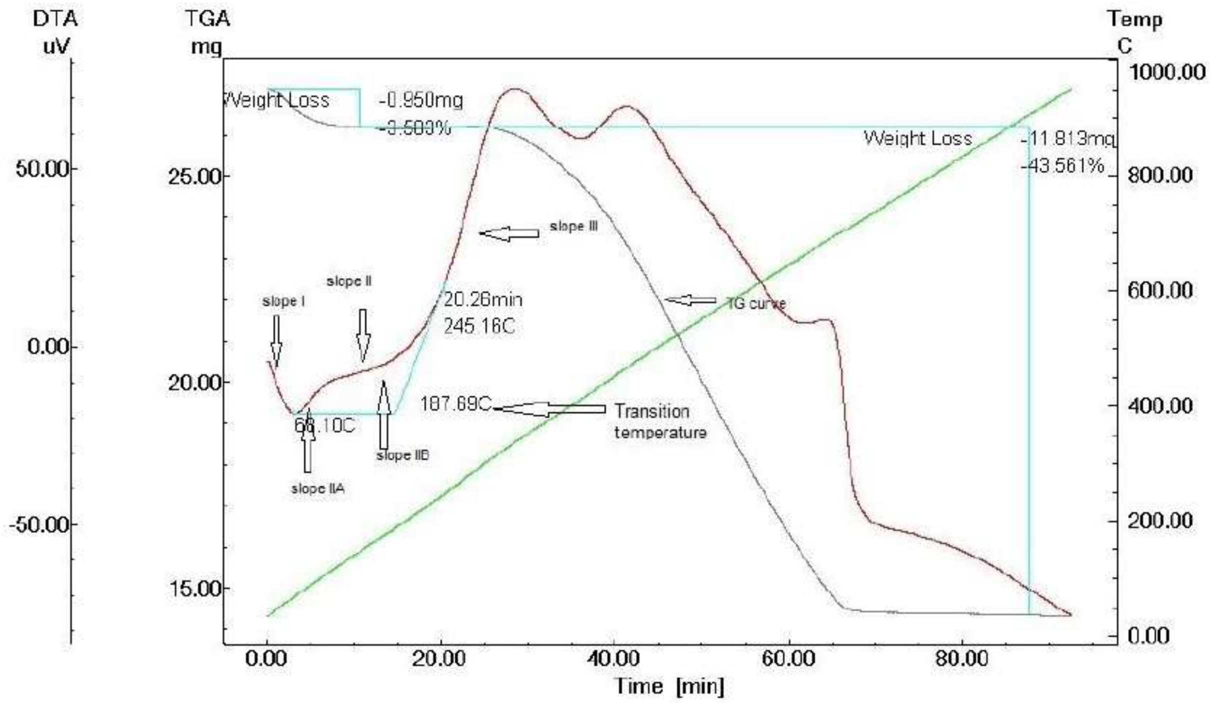

Figure 3.2. Sample DTA Graph

The DTA graph consists of three curves, namely DTA curve i.e. curve of temperature difference of sample and reference against time, TGA curve i.e. graph of change of mass of sample and the rise in temperature curve.

The DTA curve is divided into 3 parts or stages, Stage I, Stage II and Stage III.

In the initial stage i.e. stage I, endothermic reaction takes place and the loss of moisture happens. The corresponding point on the TGA curve determines the complete loss of moisture and by weight loss parameter of TGA curve the moisture content can be found out. Stage II is divided into two parts, IIA and IIB. In IIA, the heating tendency of coal starts with some residual endothermic reactions. The point of start of stage IIA is called inflexion temperature. In the second stage of Stage II, an exothermic reaction starts. The slopes of these stages i.e. IIA, IIB and overall slope of II helps in determining proneness of coal to spontaneous combustion. Lower the slopes, lower is the susceptibility of coal to spontaneous heating. The starting of stage III determines the transition temperature which plays a crucial role in determining the susceptibility of coal to spontaneous combustion. The lower the transition temperature, susceptibility to spontaneous combustion increases. After these stages, temperature rises steeply.

Procedure:

1. Size of coal sample used is -212 microns BSS weighing about 10 mg.
2. The reference material used is aluminium oxide, Al_2O_3.
3. The software is programmed to run until 450°C at a rate of 5°C per minute. After the heating is terminated, curves are obtained.

CHAPTER 4

RESULT AND ANALYSIS

Results and analysis of the experimentation are discussed below.

4.1. Proximate Analysis results

Proximate analysis of the coal samples (as received) was carried out as per IS 1350 and the results obtained are depicted in table 4.1.

Table 4.1 Result of proximate analysis

Sample.	Moisture (%)	Ash (%)	V.M. (%)	F.C. (%)
MCL-1	8.23	26.4	35.2	30.17
MCL-2	8.47	26.68	37.14	27.71
MCL-3	6.32	47.03	28.93	17.72
MCL-4	8.54	41.74	22.30	27.42
MCL-5	3.25	41.06	28.13	27.56
MCL-6	3.57	32.61	13.77	50.05
MCL-7	7.25	47.03	28.93	16.79
MCL-8	7.45	41.74	29.30	21.51
MCL-9	4.28	41.06	30.48	24.18
MCL-10	3.45	41.27	27.13	28.15
MCL-11	7.72	47.28	29.16	15.84
CCL-1	1.34	39.40	29.62	29.64
CCL-2	2.04	49.72	23.58	24.66
CCL-3	1.81	31.51	33.4	33.28
CCL-4	0.9	13.14	32.14	53.82
CCL-5	2.14	30.21	29.65	36.74
CCL-6	2.14	30.01	30.91	36.94
CCL-7	1.6	26.02	26.72	45.66
CCL-8	1.53	54.4	21.35	22.72
CCL-9	0.9	49.13	17.34	32.63

4.2. Differential thermal analysis results

Thermograms obtained from DTA of coal samples are depicted in figures 4.1 to 4.20 and the results are summarized in table 4.2

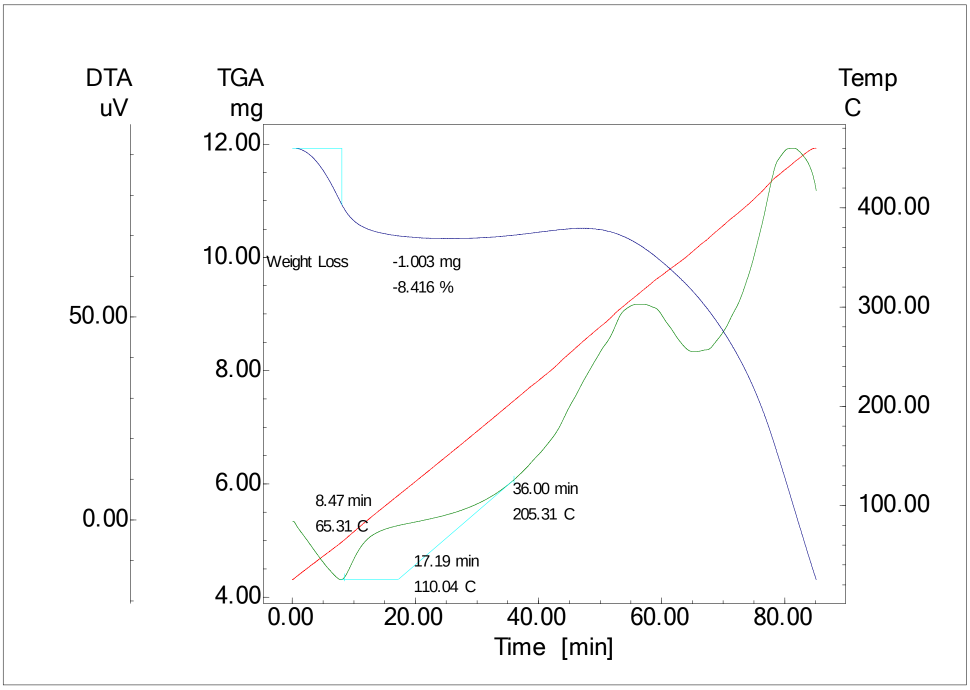

Figure 4.1. DTA-TG Curve of MCL-1 sample

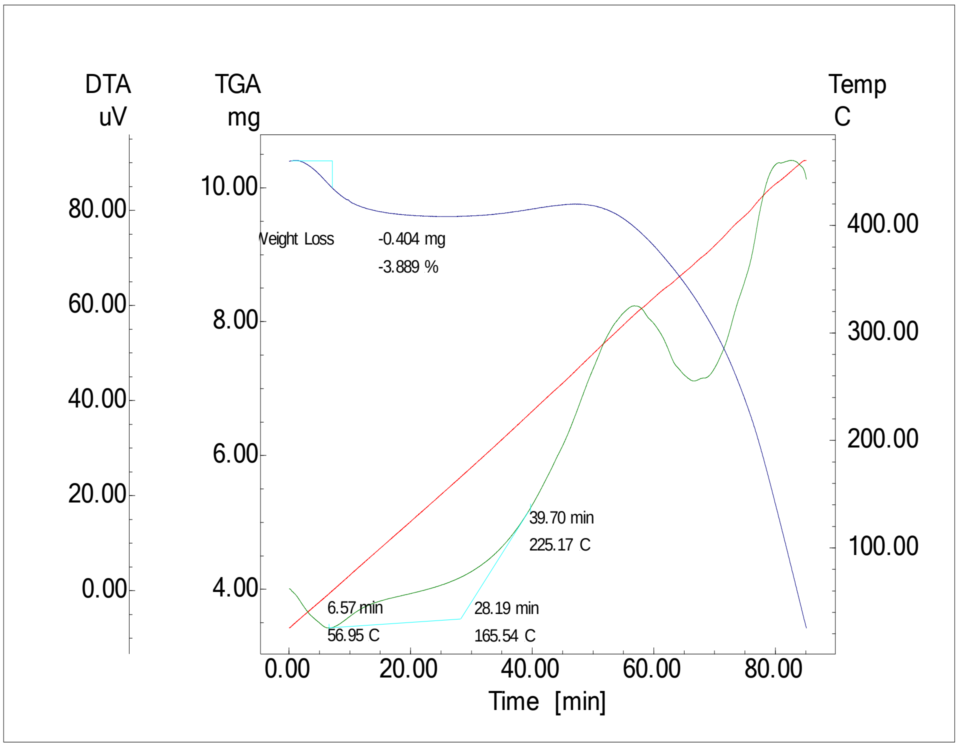

Figure 4.2. DTA-TG Curve of MCL-2 sample

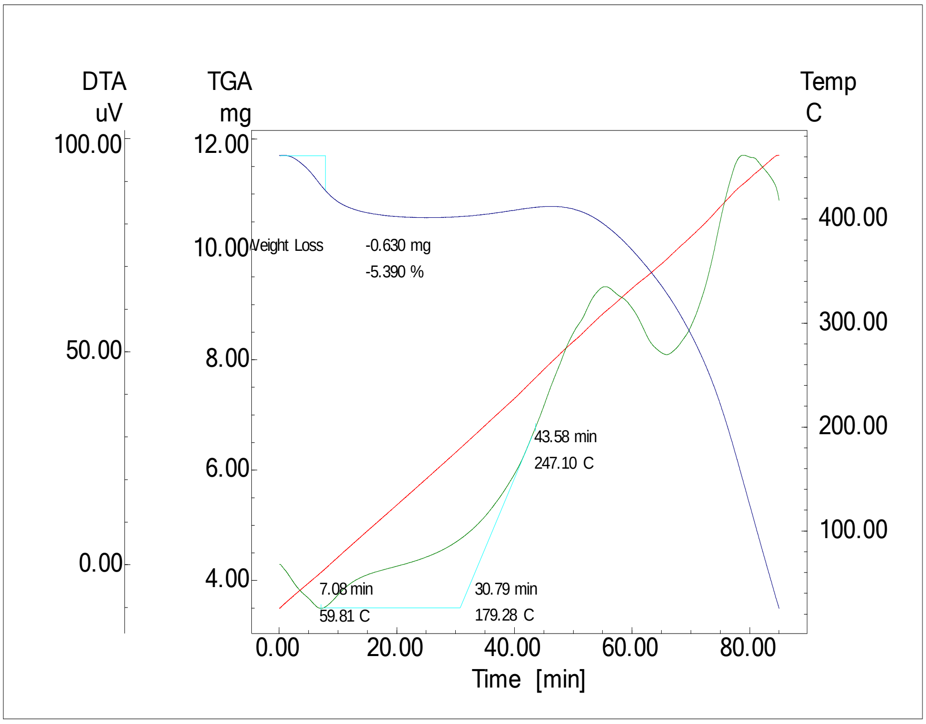

Figure 4.3. DTA-TG Curve of MCL-3 sample

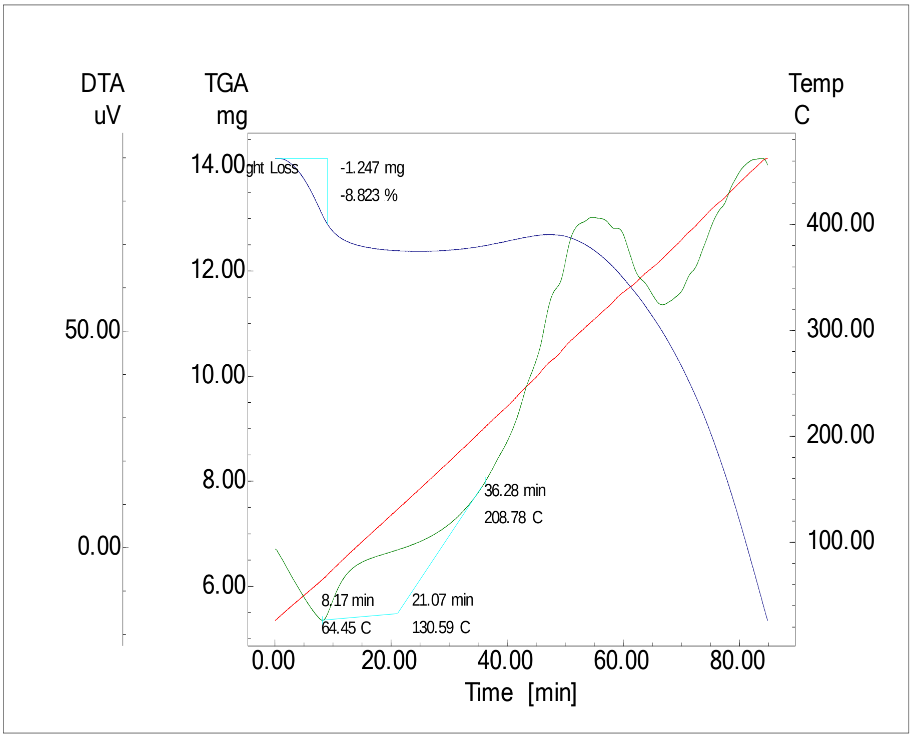

Figure 4.4. DTA-TG Curve of MCL-4 sample

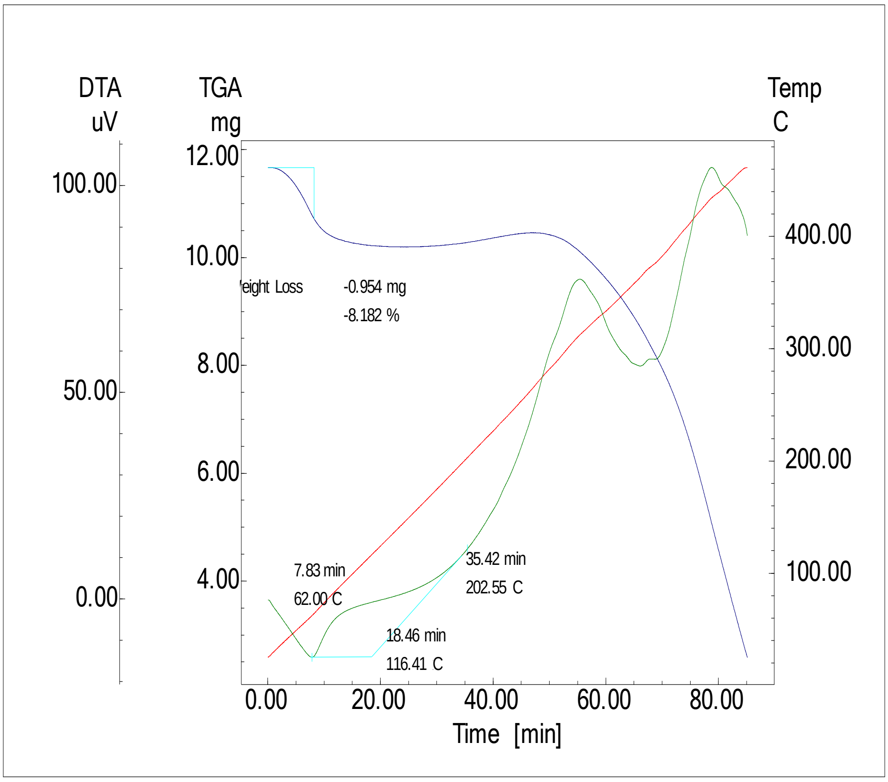

Figure 4.5. DTA-TG Curve of MCL-5 sample

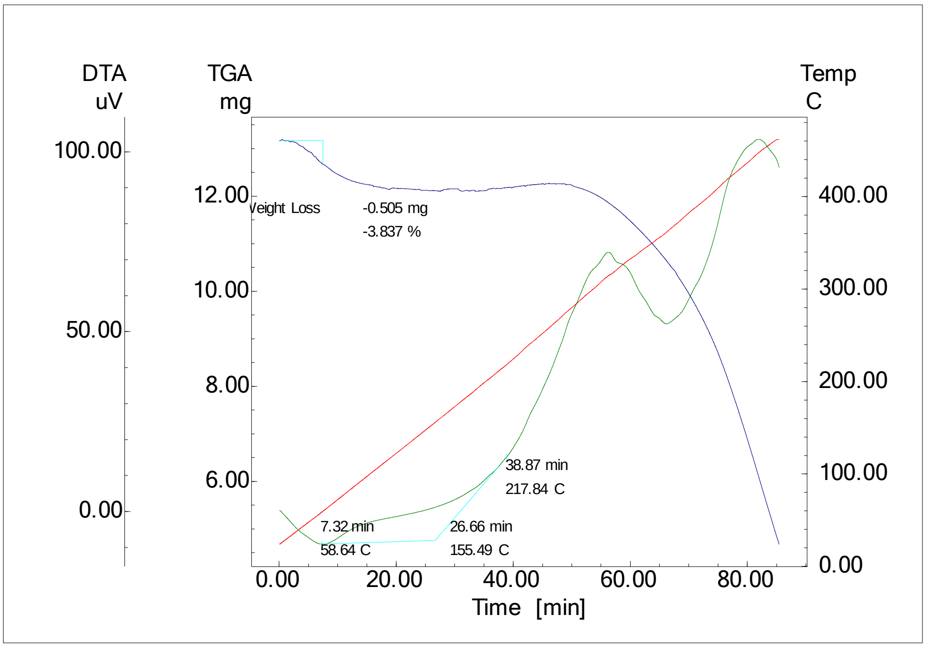

Figure 4.6. DTA-TG Curve of MCL-6 sample

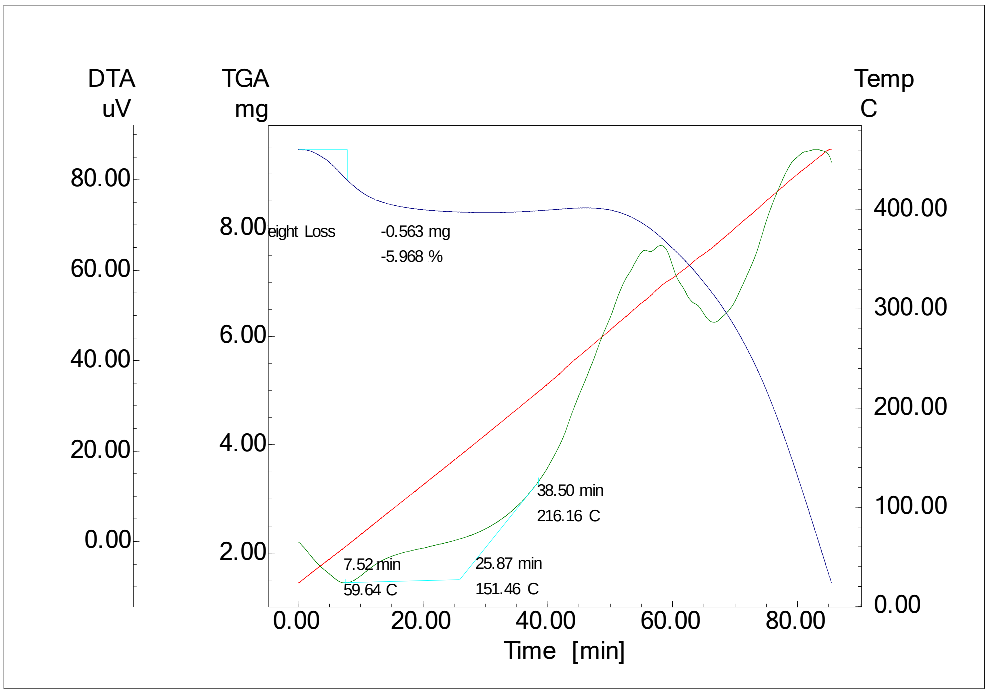

Figure 4.7. DTA-TG Curve of MCL-7 sample

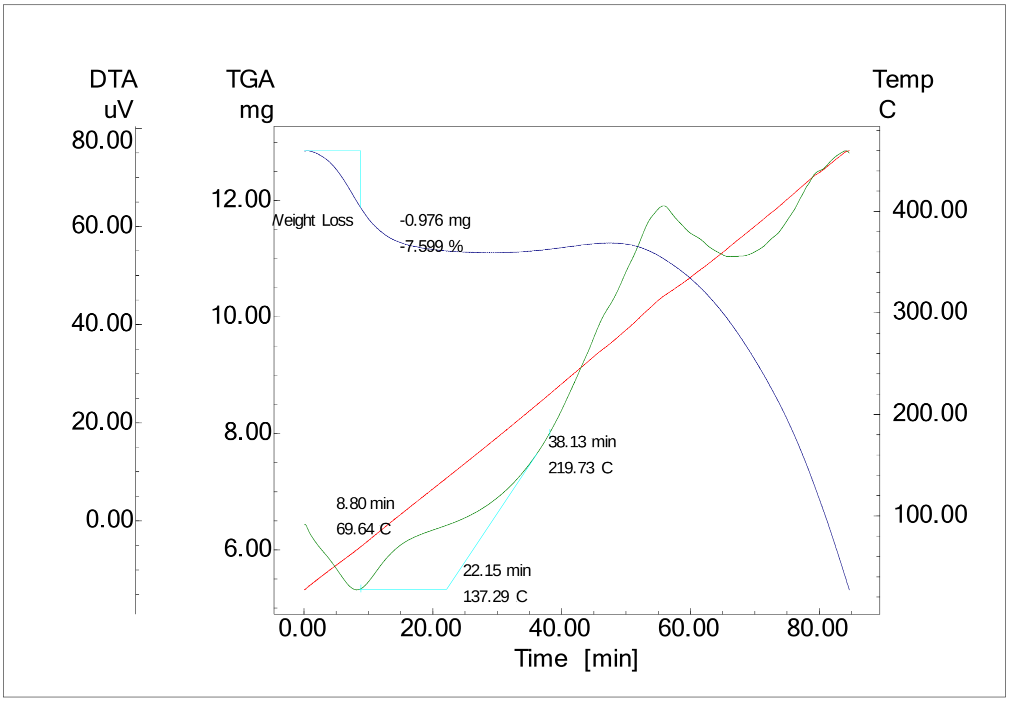

Figure 4.8. DTA-TG Curve of MCL-8 sample

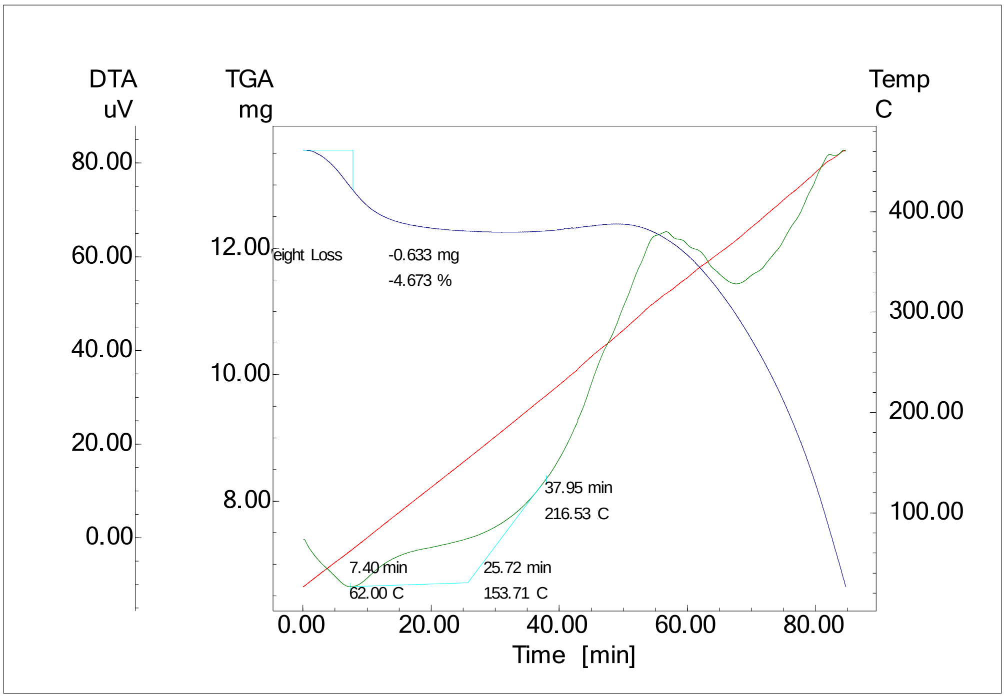

Figure 4.9. DTA-TG Curve of MCL-9 sample

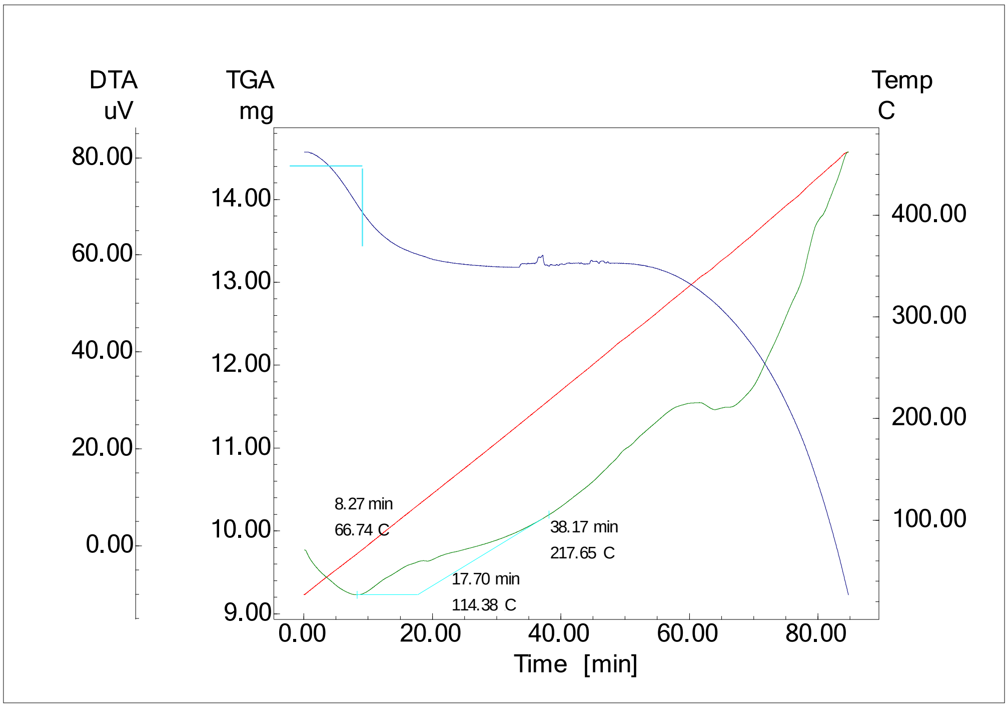

Figure 4.10 DTA-TG Curve of MCL-10 sample

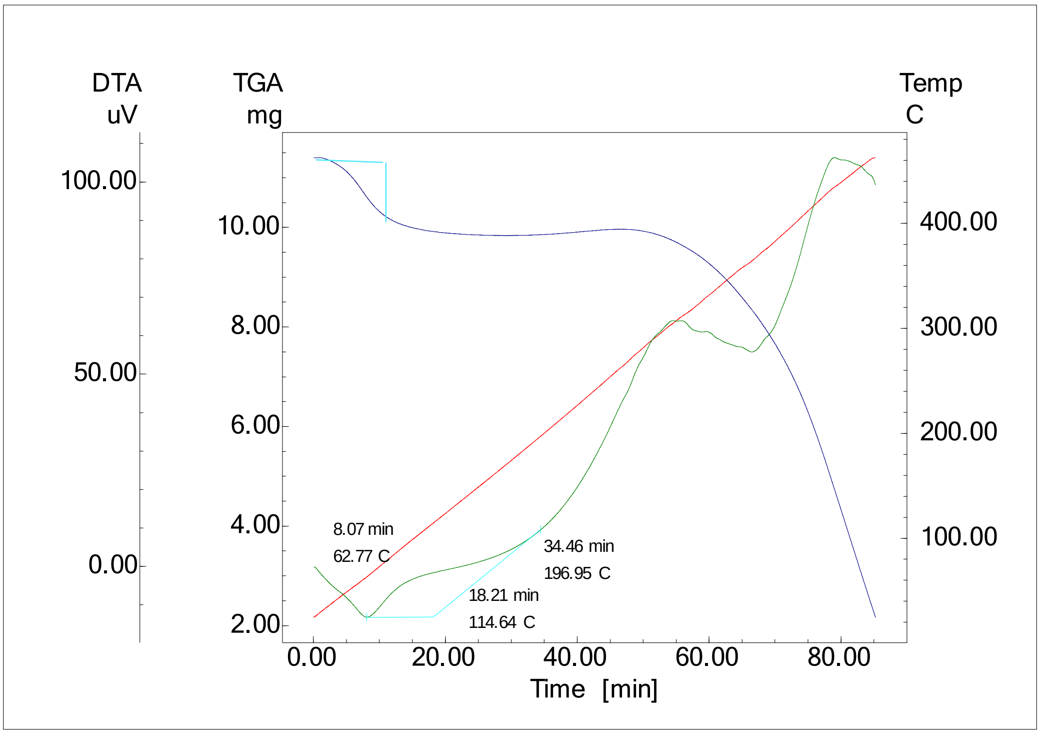

Figure 4.11. DTA-TG Curve of MCL-11 sample

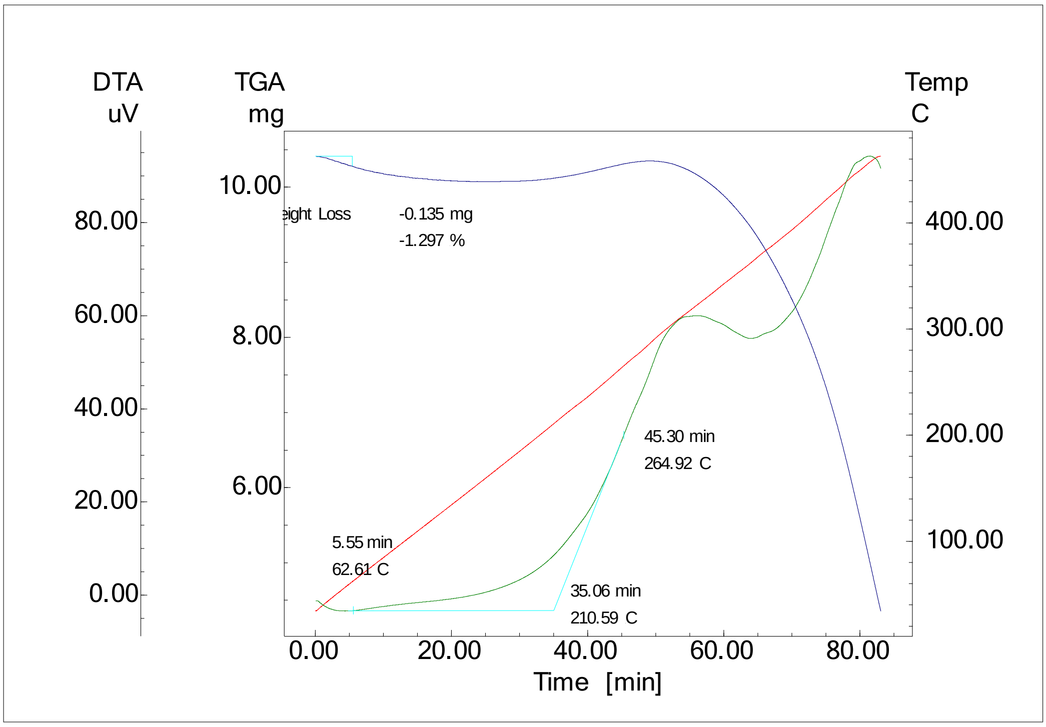

Figure 4.12. DTA-TG Curve of CCL-1 sample

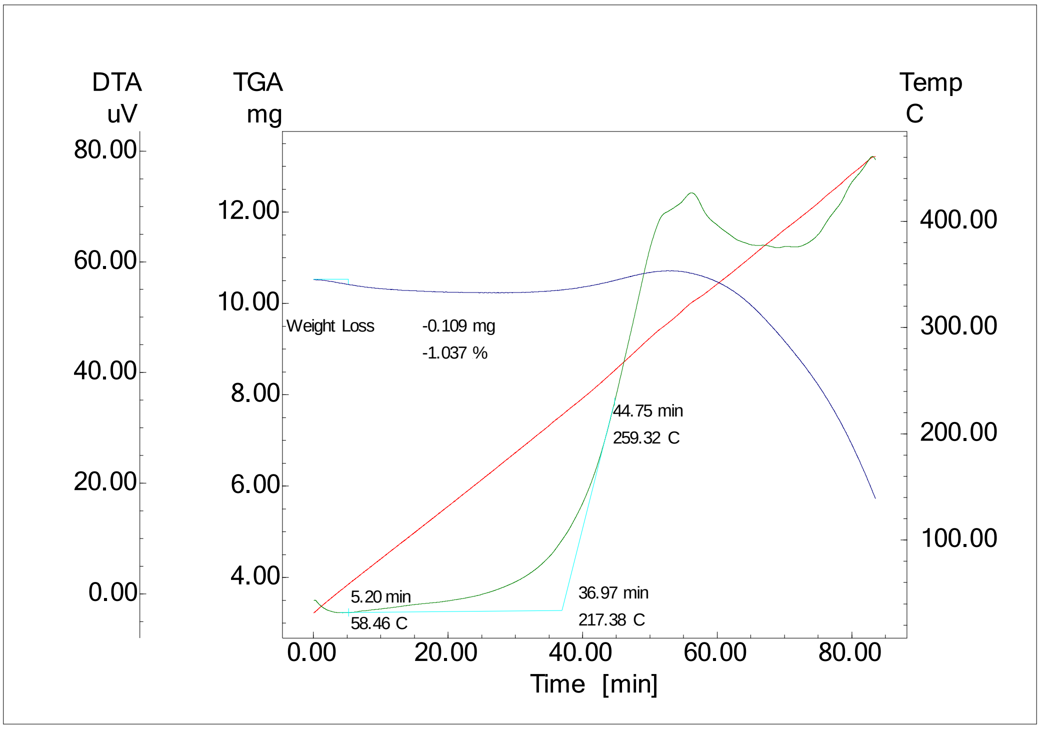

Figure 4.13. DTA-TG Curve of CCL-2 sample

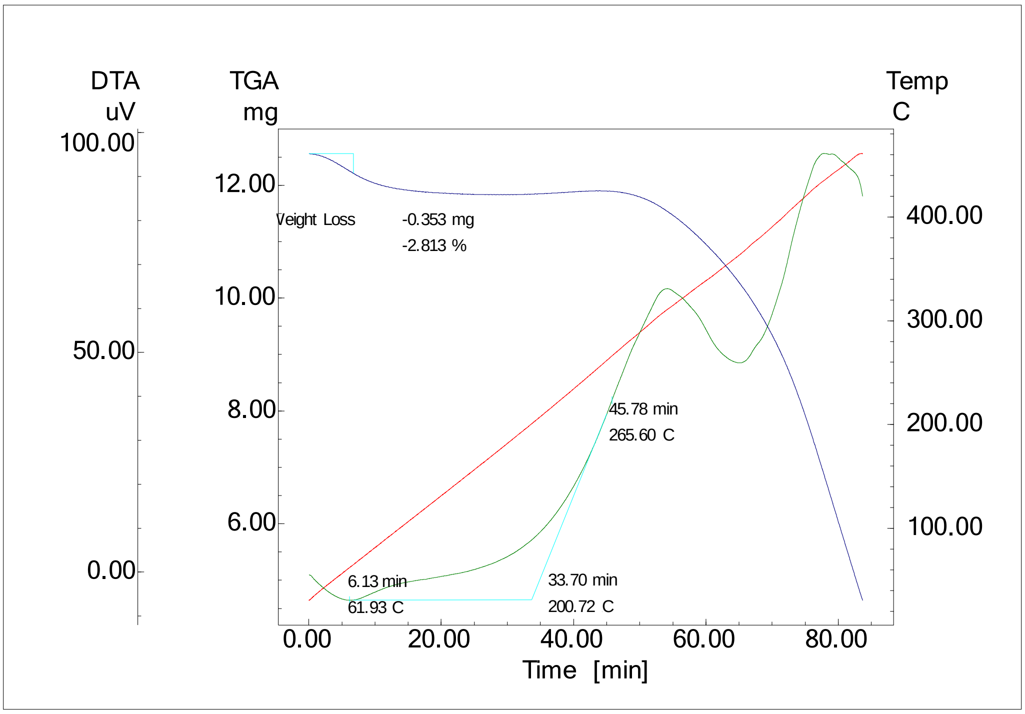

Figure 4.14. DTA-TG Curve of CCL-3 sample

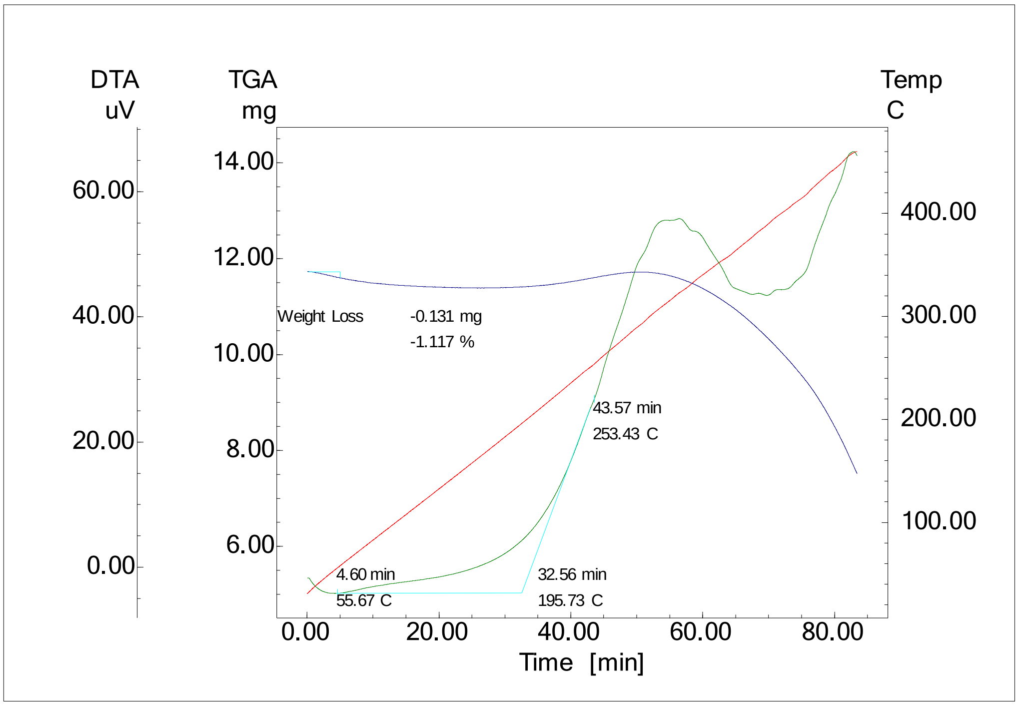

Figure 4.15. DTA-TG Curve of CCL-4 sample

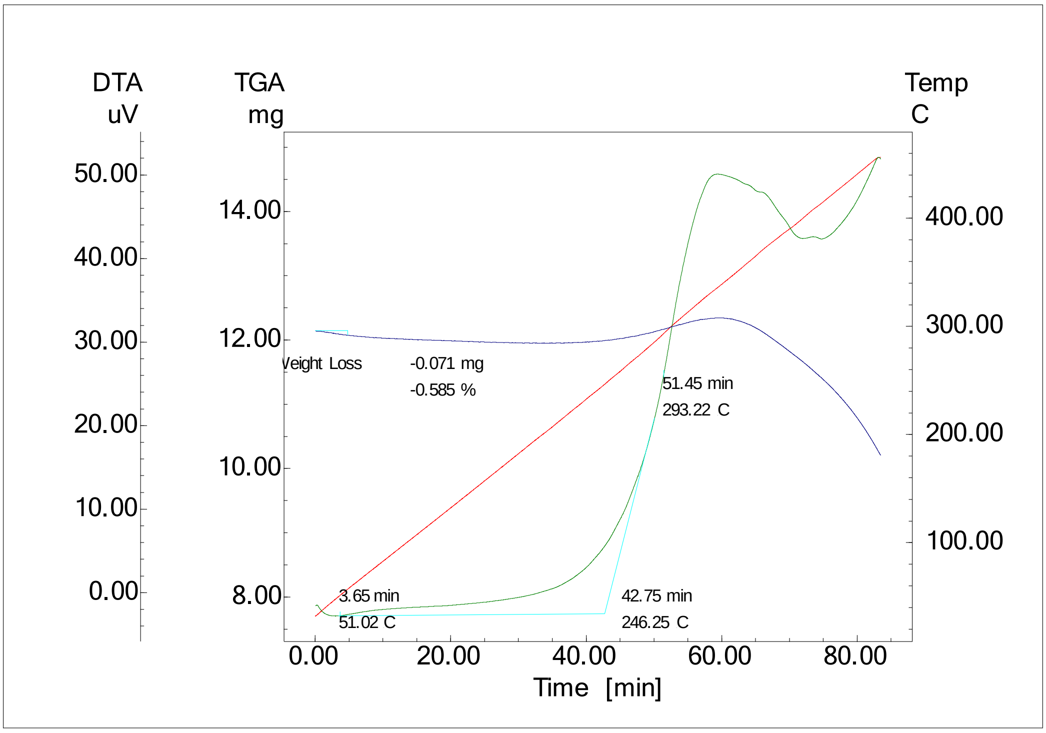

Figure 4.16. DTA-TG Curve of CCL-5 sample

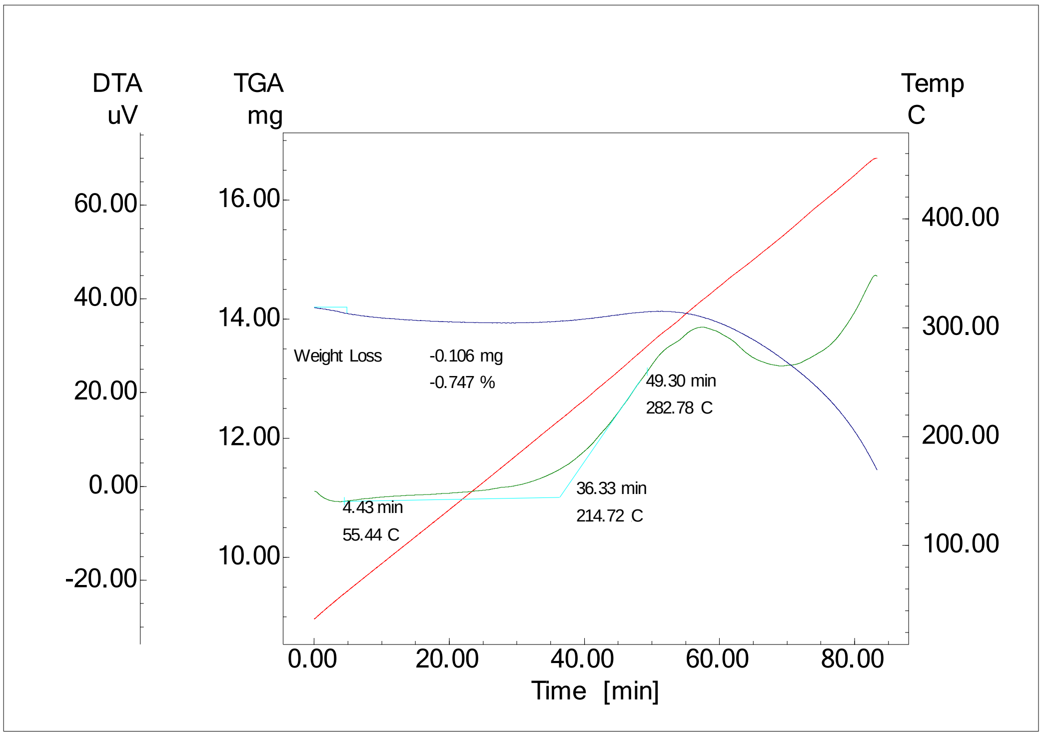

Figure 4.17. DTA-TG Curve of CCL-6 sample

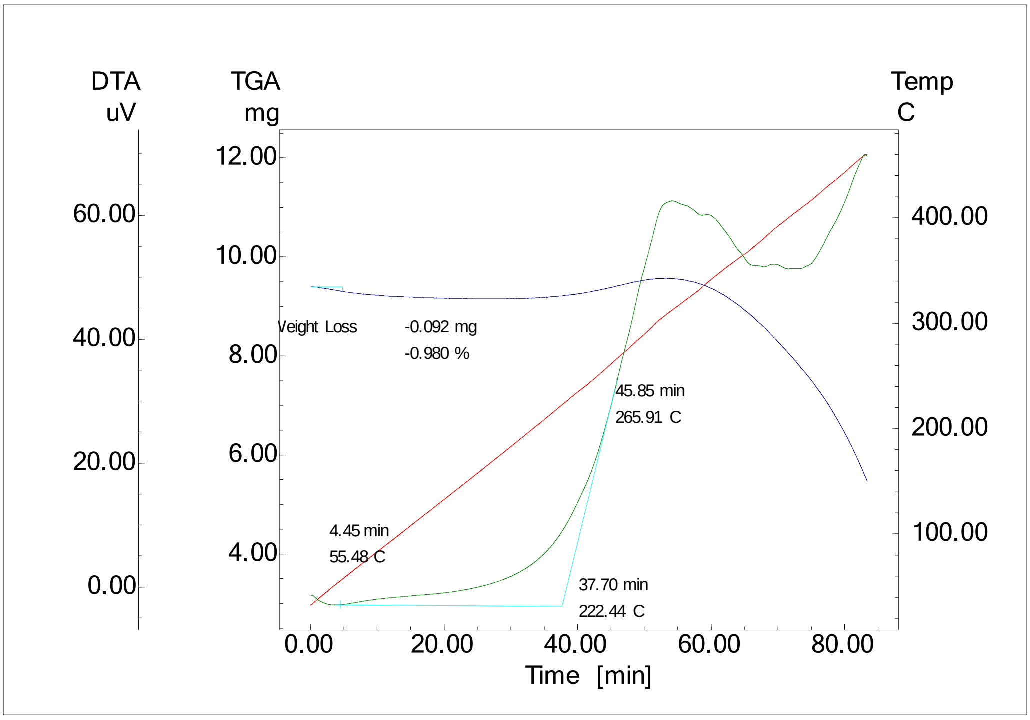

Figure 4.18. DTA-TG Curve of CCL-7 sample

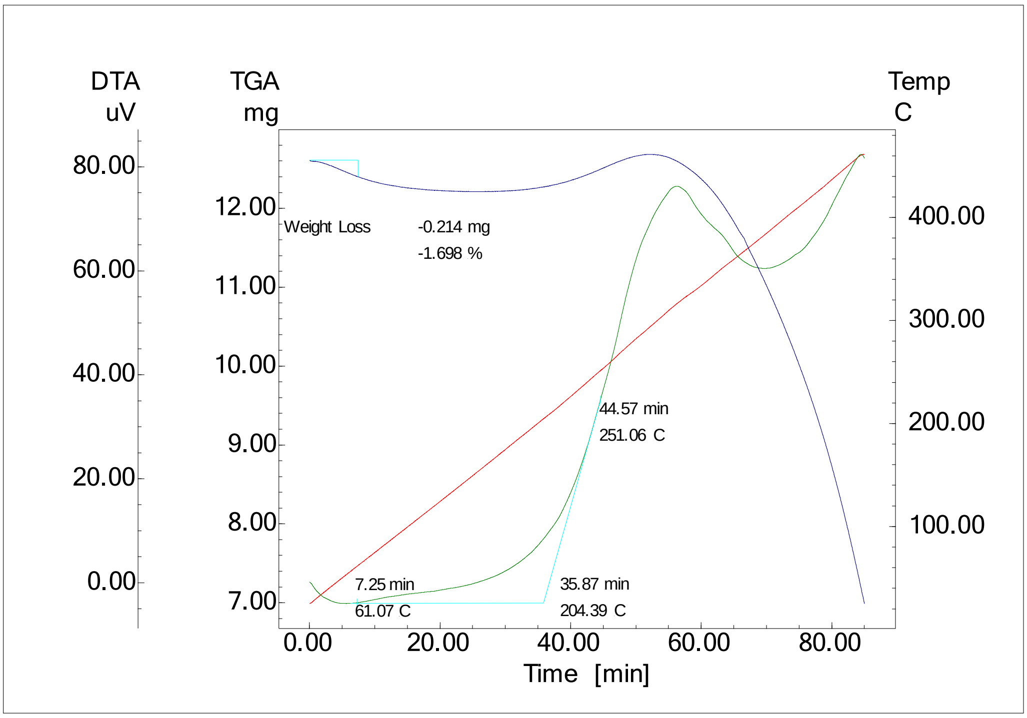

Figure 4.19. DTA-TG Curve of CCL-8 sample

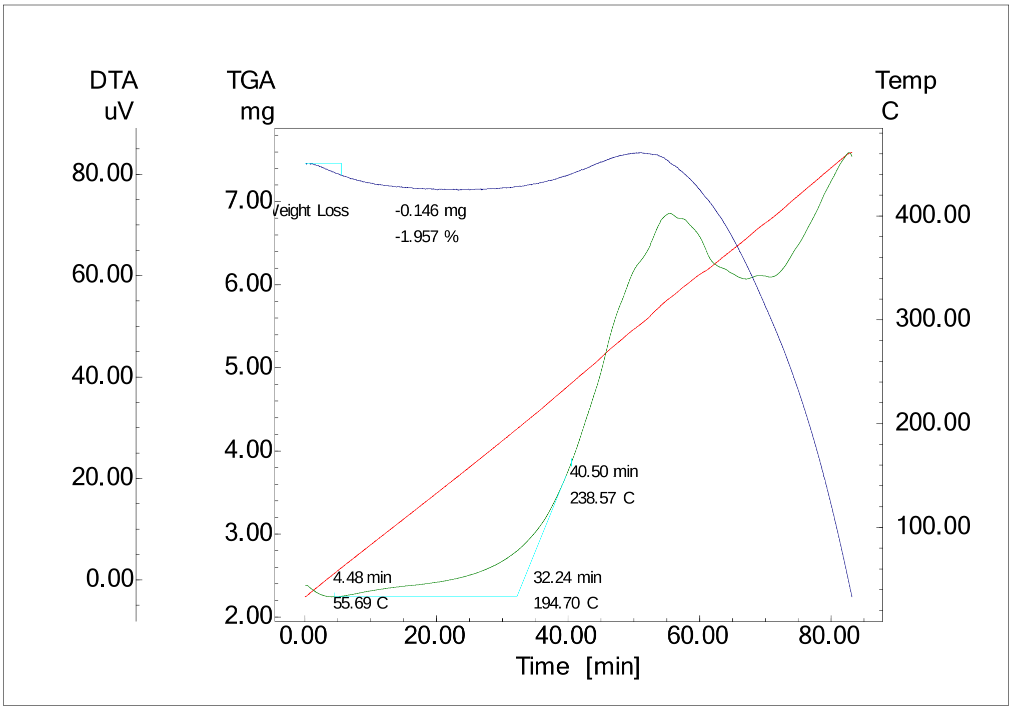

Figure 4.20. DTA-TG Curve of CCL-9 sample

4.3. Observation from DTA curve

Table 4.2 Observation from DTA Thermogram

Samples	Slope IIA	Slope IIB	Slope II	Transition Temperature (°C)
MCL-1	0.28482	0.1182	0.17144	110.04
MCL-2	0.07073	0.24367	0.13203	165.54
MCL-3	0.1361	0.38573	0.22649	179.28
MCL-4	0.24569	0.20297	0.22255	130.59
MCL-5	0.24521	0.14906	0.18625	116.41
MCL-6	0.10563	0.22406	0.15201	155.49
MCL-7	0.14823	0.2442	0.1879	151.46
MCL-8	0.12491	0.18219	0.15637	137.29
MCL-9	0.10152	0.10793	0.10412	153.71
MCL-10	0.23006	0.17817	0.19455	114.38
MCL-11	0.29227	0.15721	0.20942	114.64
CCL-1	0.08075	0.47248	0.18595	210.59
CCL-2	0.08224	0.5949	0.18929	217.38
CCL-3	0.09914	0.4869	0.22266	200.72
CCL-4	0.06019	0.39185	0.15696	195.73
CCL-5	0.04277	0.43688	0.1192	246.25
CCL-6	0.04282	0.30591	0.12158	214.72
CCL-7	0.07122	0.55119	0.17037	222.44
CCL-8	0.08561	0.57253	0.20522	204.39
CCL-9	0.06518	0.39708	0.14479	194.70

4.4. Correlation Analysis

The regression analysis was carried out between the results of DTA analysis and the constituents of proximate analysis using MS Excel and the correlation coefficients obtained from it are depicted in table 4.3.

Table 4.3. Correlation analysis between DTA and proximate analysis

	Slope IIA	Slope IIB	Slope II	Transition Temperature
Moisture	0.80673	-0.72613	0.309326	-0.806804713
Ash	0.01867	0.121274	0.276283	-0.046836521
Volatile Matter	0.291272	-0.26578	0.010152	-0.291684916
Fixed Carbon	-0.32481	0.175874	-0.36156	0.363984093

It is found form the table that for determining the susceptibility of coal samples to spontaneous combustion, moisture content shows good correlation with transition temperature (r = -0.807); ash content shows competitive relation with Slope II value (r = 0.276); volatile matter shows good correlation with transition temperature (r = -0.292) and fixed carbon content with transition temperature (r = 0.363). For correlation analysis, slope II and transition temperature are plotted against moisture, volatile matter, ash and fixed carbon content and regression analysis wass done using Microsoft excel 2013.

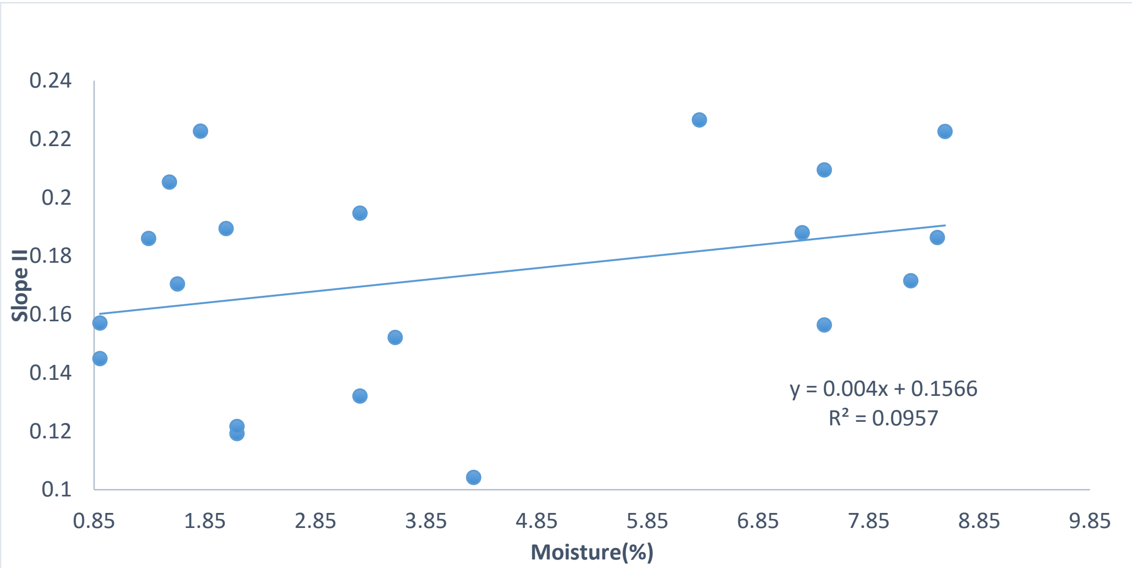

Figure 4.21. Correlation between moisture and slope II obtained from DTA curve

It is observed that with an increase in moisture content of the coal sample, the slope of stage II also increases, suggesting both are directly proportional.

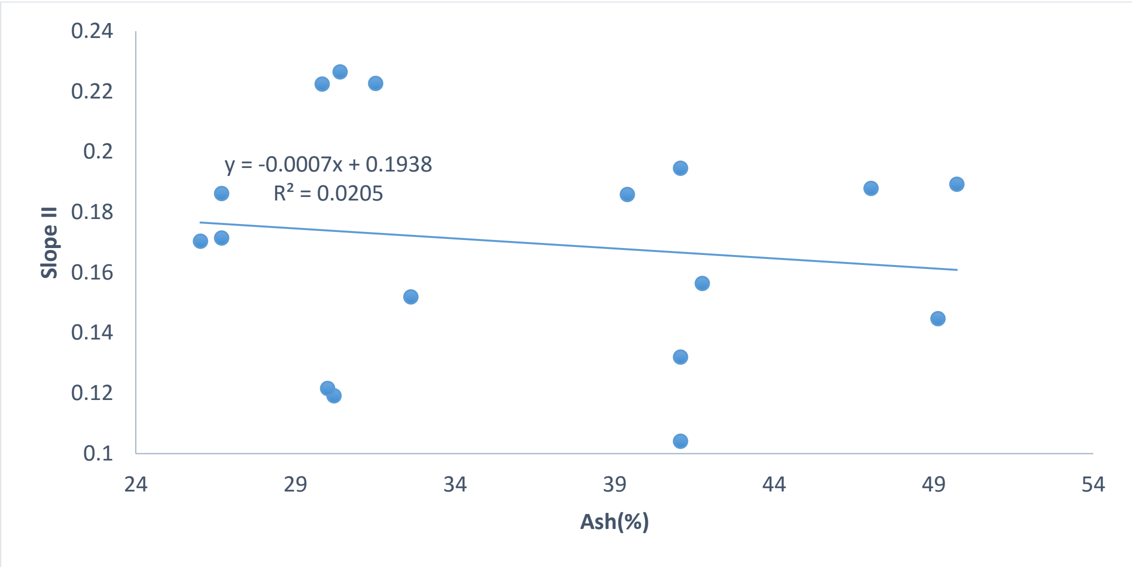

Figure 4.22. Correlation between ash and slope II obtained from DTA curve

It is observed that with an increase in ash content of the coal sample, the slope of stage II decreases, suggesting both are indirectly proportional.

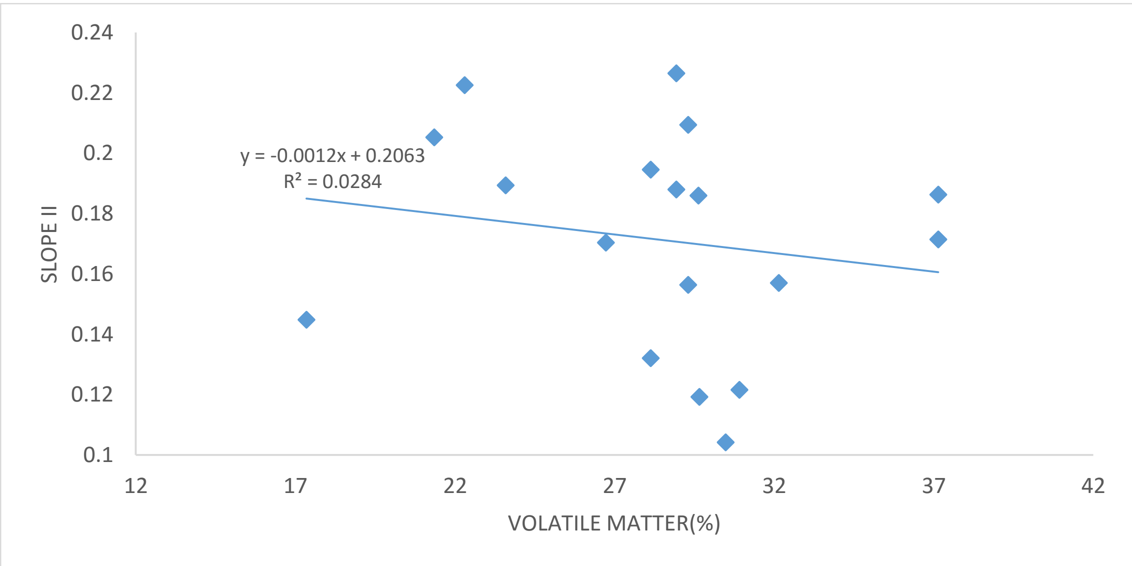

Figure 4.23. Correlation between volatile matter and slope II obtained from DTA curve

It is observed that with an increase in volatile matter content of the coal sample, the slope of stage II decreases, suggesting both are inversely proportional.

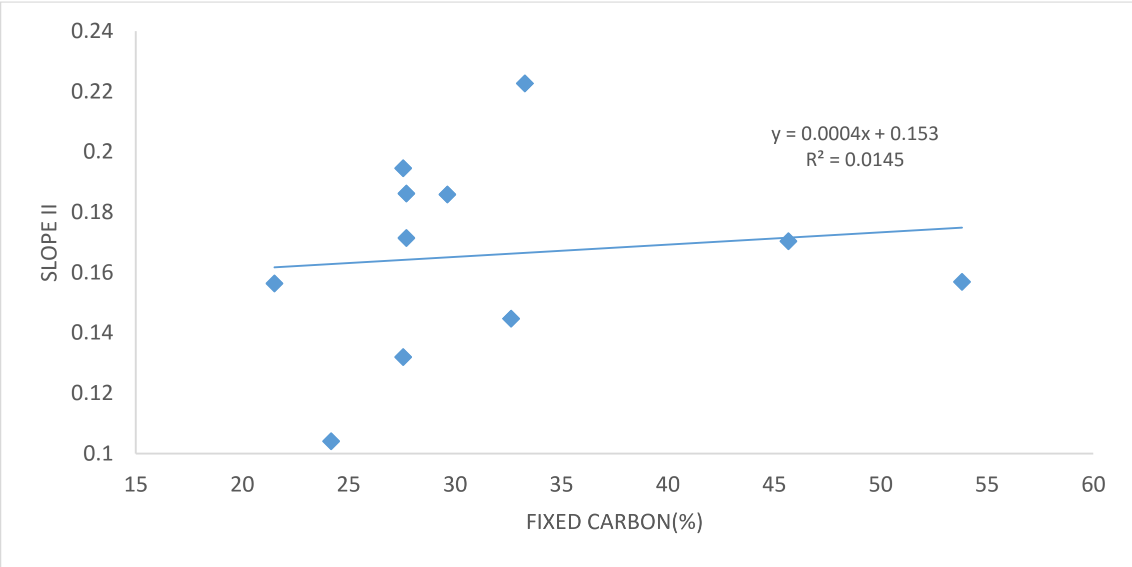

Figure 4.24. Correlation between fixed carbon and slope II obtained from DTA curve

It is observed that with an increase in fixed carbon content of the coal sample, the slope of stage II also increases, suggesting both are directly proportional.

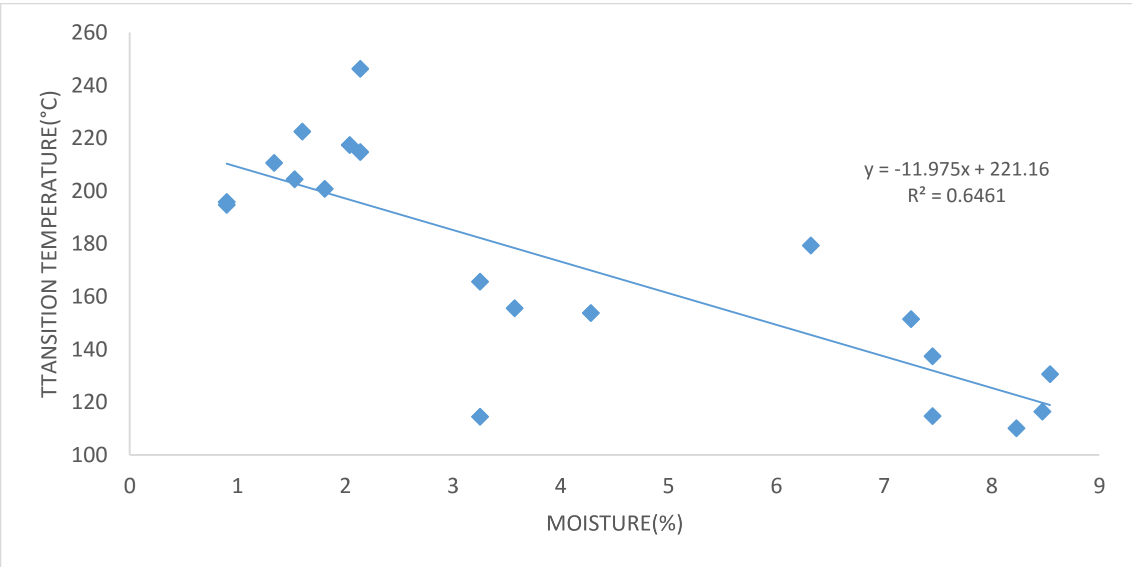

Figure 4.25. Correlation between moisture and transition temperature

It is observed that with an increase in moisture content of the coal sample, the transition temperature decreases, suggesting both are inversely proportional.

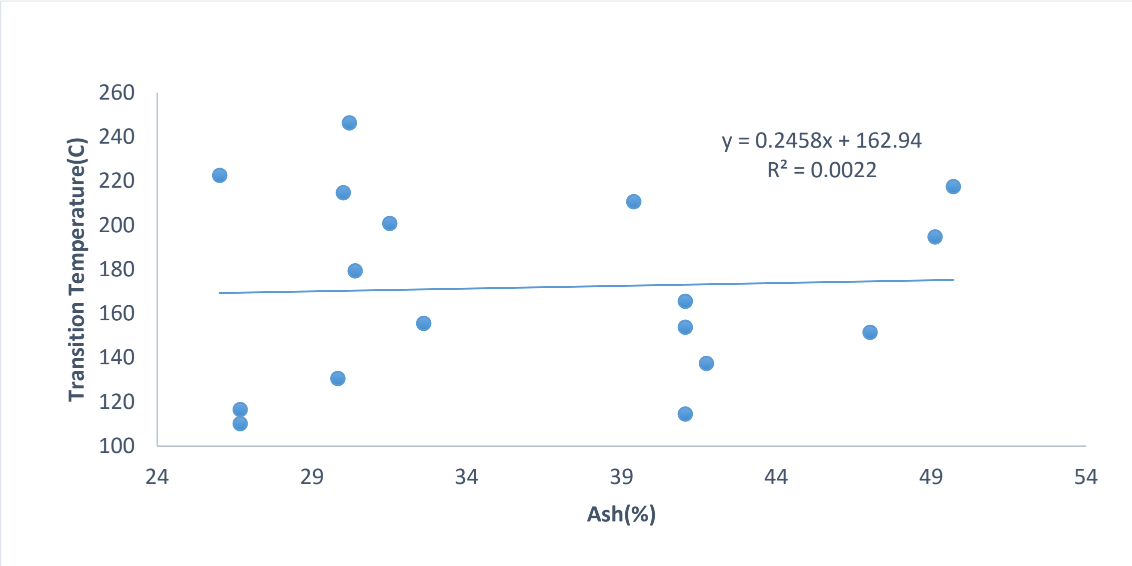

Figure 4.26. Correlation between ash and transition temperature

It is observed that with an increase in ash content of the coal sample, the transition temperature also increases, suggesting both are directly proportional.

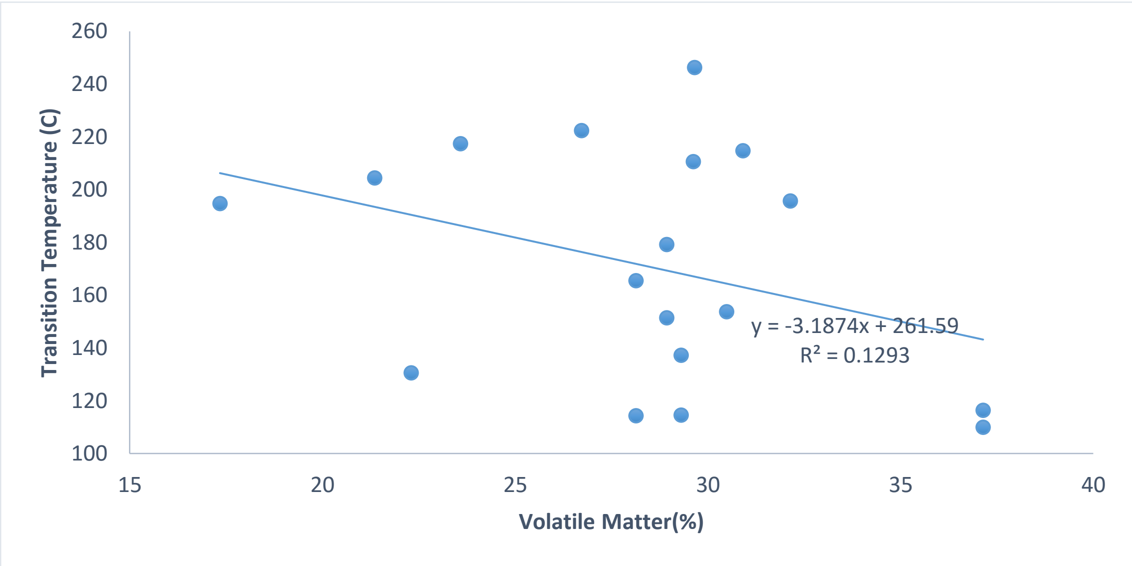

Figure 4.27. Correlation between volatile matter and transition temperature

It is observed that with an increase in volatile matter content of the coal sample, the transition temperature decreases, suggesting both are inversely proportional.

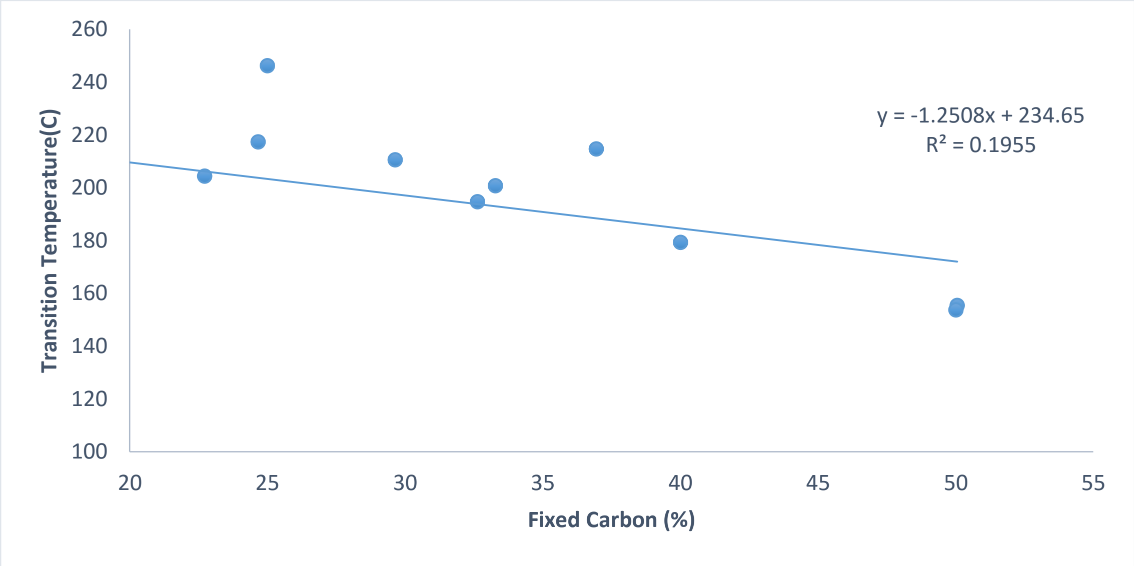

Figure 4.28. Correlation between fixed carbon content and transition temperature

It is observed that with an increase in fixed carbon content of the coal sample, the transition temperature decreases, suggesting both are inversely proportional.

Chapter 5

Conclusions

Investigations of the proneness of Indian coals to spontaneous combustion using DTA was carried out and the findings were correlated with the proximate analysis, i.e., with moisture, ash, volatile matter and fixed carbon content. The correlation was carried out using Microsoft Excel 2013 by finding out the correlation coefficient between various constituents of proximate analysis and the parameters of DTA.

From the results and analysis, the coal samples collected from the mines can be identified into poorly susceptible, moderately susceptible and highly susceptible to spontaneous combustion in the following table.

Sl. No.	Susceptibility to spontaneous combustion	Coal Samples
1	Highly	MCL-1, MCL-5, MCL-4, MCL-8, MCL-10, MCL-11
2	Moderately	MCL-2, MCL-6, MCL-7, MCL-9
3	Poorly	MCL-3, CCL-1, CCL-2, CCL-3, CCL-4, CCL-5, CCL-6, CCL-7, CCL-8, CCL-9

It is also concluded that from the thesis work carried out that:

- Increase in moisture content leads to increase in proneness of coal to auto oxidation (regression equation with transition temperature as $y = -11.975x+221.16$ with $R^2 = 0.646$).
- Increase in ash content leads to decrease in spontaneous combustion of coal (regression equation with. Slope II as $y = -0.0007x + 0.1938$ with $R^2 = 0.0205$).
- With increase in volatile matter content, the susceptibility to spontaneous combustion increases (regression equation with transition temperature as $y= -3.1874x + 261.59$ with $R^2 = 0.1293$).

- With increase in fixed carbon content the susceptibility to spontaneous heating decreases (regression equation with transition temperature as y = -1.2508x + 234.65 with R^2 = 0.1955).

References

1. Nimaje D.S, Tripathy D.P., “Thermal studies on spontaneous heating of coal”, The Indian Mining & Engineering Journal, pp. 10 – 21, 2010.
2. www.industry.gov.au/Office-of-the-Chief.../Coal-in-India.pdf (Coal in India 2015).
3. en.wikipedia.org/wiki/coal seam fire
4. en.wikipedia.org/wiki/spontaneous-combustion
5. Ramlu M.A., Mine Disaster and Mine Rescue, Capter-1, Oxford and IBH publishing Co Pvt Ltd, 2nd edition, pp. 10-20, 1991.
6. Banerjee S.C., Spontaneous Heating of coal and Mine Fires, Oxford and IBH publishing Co Pvt Ltd, 1st edition, pp.1-40, 1985.
7. Sahu H.B., Panigrahi D.C. and Mishra N.M., “Assessment of spontaneous heating susceptibility of coal seams by experimental techniques – a complete study”, International Symposium on advances in mining technology and management, IIT Kharagpur, 2005, pp. 459-465.
8. Mohalik N. K. and Singh V. K., “Application of thermal analysis techniques to assess proneness of coal to spontaneous heating”, Journal of Thermal Analysis and Calorimetry”, vol. 98, no. 2, pp. 507-519, 2009. ISSN 1388-6150.
9. Saikia B.K., Boruah R.K., Gogoi P.K. and Baruah B.K., “A Thermal Investigation of coals from Assam”, Journal of Fuel Processing Technology, vol. 90, issue 2, pp. 196-203, 2009.
10. Nimaje D.S., Tripathy D.P., “Characterisation of some Indian coals to assess their liability to spontaneous combustion”, Fuel, vol. 161, pp 139-147, 2016.
11. Pattanaik D. S., Behera P. and Singh B., “Spontaneous combustibility characterisation of the Chirimiri coals, Koriya District, Chhatisgarh, India”, International Journal of Geosciences, vol. 2, pp. 336-347, 2011.
12. Gouwls M.J., Wade L., “Self-heating liability of coal”, Mining Science and Technology, vol. 9, issue 1, pp. 81-85, 1989.
13. Clemens A. H., Matheson T. W. and Rogers D. E., “DTA studies of the low temperature oxidation of low rank coals”, Fuel, vol. 69, pp. 255-256, 1990.
14. Pis J.J., Puente G., Fuente E., Moran A., Rubiera F., “A study of self-heating of fresh and oxidised coal using differential thermal analysis”, Thermochimica Acta, vol 279, pp. 99-103, 1996.

15. Jain A. (2009), "Assessment of spontaneous heating of coal differential thermal analysis", B.Tech thesis, pp.1-25
16. Deshmukh D.J.(2011), Elements of Mining Technology Vol 2, Denett and Co. Publications, pp. B-1.25
17. I.S. (Indian Standard): 436 (Part I, Sec. 1) – 1964, Method for sampling of coal and coke: sampling of coal, Manual sampling, Bureau of Indian Standards, New Delhi
18. I.S. (Indian Standard) (1350) (Part-I) – 1984, Methods of Test for Coal and Coke: Proximate Analysis, Bureau of Indian Standards, New Delhi, pp.3-28.